

**EFFECT OF PHOSPHINE  
AGAINST  
THE PINK BOLLWORM  
IN  
BAGGED COTTONSEED**

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# EFFECT OF PHOSPHINE AGAINST THE PINK BOLLWORM IN BAGGED COTTONSEED

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## SUMMARY

Phosphine from aluminum phosphide tablets was successfully used in fumigating larvae of the pink bollworm (*Pectinophora gossypiella* (Saunders)) in stacks of bagged cottonseed. Studies were conducted at Mesa, Ariz., Manhattan, Kans., and Chickasha, Okla., during 1968 and 1969. Both laboratory-reared pink bollworm larvae and larvae in cotton bolls in the field were used.

In the Mesa study all the pink bollworm larvae were killed after an exposure of 120 hours to fumigant evolved from 180 aluminum phosphide tablets per 1,000 cubic feet. The temperature of the cottonseed at the start of the test was 59° F. and the relative humidity in the warehouse was 62 percent. During exposure the warehouse temperature ranged from 45° to 66° and the relative humidity within the tarped stacks from 50 to 62 percent. Germination tests showed that two fumigations with 180 aluminum phosphide tablets per 1,000 cubic feet had no effect on the viability of the cottonseed.

At Manhattan additional laboratory studies indicated the dosage could be reduced substantially.

At the Oklahoma Cotton Research Center at Chickasha, field studies showed that 60 aluminum phosphide tablets per 1,000 cubic feet in a 120-hour exposure would release sufficient phosphine gas ( $\text{PH}_3$ ) to penetrate and kill all pink bollworm larvae in field-infested cotton bolls and seeds within commercial cottonseed shipping bags covered with a 1.5-mil polyethylene tarp. The temperature was 42° F. and the relative humidity 60 percent when the fumigant was applied. The temperature ranged from 36° to 53° and the relative humidity from 30 to 85 percent during the test.

The findings of these studies were used to establish a schedule in the regulatory program of the Plant Protection Division, Agricultural Research Service, to permit movement of cottonseed out of pink bollworm quarantine areas.

## INTRODUCTION

Cooperative fumigation studies were conducted to determine the dosage rate of aluminum phosphide tablets and the exposure time necessary to kill 100 percent of the larvae of the pink

bollworm (*Pectinophora gossypiella* (Saunders)) in cottonseed. The work was done at Mesa, Ariz., Manhattan, Kans., and Chickasha, Okla.

## TESTS IN ARIZONA

The first two tests were conducted at Mesa, where Arizona Cotton Planting Seed Distribu-

tors made available cottonseed and warehouse facilities.

## Test 1

### Materials and Methods

Five stacks of cottonseed were set up for fumigation in a 60- by 120-foot warehouse. Thirty-six cotton fiber bags, each containing 50 pounds of cottonseed, were placed on 4- by 5-foot pallets. Each stack was two pallets square and three pallets high and contained about 1,000 cubic feet.

Laboratory-reared pink bollworm larvae and larvae in field-infested cotton bolls were used. To prevent cannibalism among the laboratory-reared larvae, a small amount of cotton fiber was inserted in small plastic vials along with some synthetic media. Then five larvae were introduced into each and covered with more cotton. A perforated cap with a 60-mesh screen insert was placed on each vial. Five vials were placed in an 8-ounce paper cup, covered with masking tape, and put in a 5- by 11-inch cotton duck sack with tie strings. One sack was placed between the bags on each of the 12 pallets of each stack. Polyethylene tubing, one-fourth inch in outside diameter, was attached to each sack containing the test insects to obtain gas samples for chromatography analyses.

Light cotton cloth sacks, 6 by 11 inches, with tie strings, were filled with pink bollworm-infested cotton bolls and placed near the bottom, middle, and top of the stacks of cottonseed. Infested cotton bolls and laboratory-reared larvae were held under similar conditions as controls. Samples of seed were taken for germination tests.

The stacks were covered with 6-mil nylon tarps. Sand snakes were laid on three sides of the stacks, aluminum phosphide tablets were put on paper plates and placed under the stacks, and sand snakes were then laid on the fourth side of the stacks. A 50-pound bag of cottonseed was placed at each corner and over the locations where the polyethylene tubing came out of the stacks to hold down the tarps and seal any openings.

The dosage rates of aluminum phosphide tablets used in the test were 60, 90, 120, 150, and 180 per 1,000 cubic feet. Gas samples were drawn at 12, 24, 48, 72, and 96 hours after placement of the tablets. The tarps were removed after 96 hours' exposure. The warehouse was aerated by natural draft for about 30 minutes, after which

the test insects and gas sampling lines were removed.

The moisture content of the fumigated cottonseed was about 5 percent. The temperature of the cottonseed at the time of fumigation was 56° F. The relative humidity in the warehouse when the stacks were tarped was 32 percent. The temperature in the warehouse ranged from 44° to 75° and the relative humidity from 25 to 84 percent. The temperature within the tarped stacks ranged from 52° to 56° and the relative humidity from 32 to 36 percent.

Mortality counts were made on the laboratory-reared pink bollworm larvae as soon as possible after they were removed from the stack. After the larvae had been examined, they were placed in small plastic boxes and held at about 60-percent relative humidity and 85° to 90° F. for 3 days before the final mortality count was made. The infested cotton bolls used in the test were also incubated for 3 days before they were examined. About 900 seeds from 30 infested cotton bolls taken from the bottom, middle, and top of each stack were split and examined. The numbers of live and dead larvae were recorded. The fumigated cotton bolls that were not dissected and examined and the controls were subjected to a process that forced any surviving larvae out of diapause.

### Results

All laboratory-reared pink bollworm larvae in the stacks fumigated with 60, 90, 150, and 180 aluminum phosphide tablets per 1,000 cubic feet were dead. One larva was found alive in the samples from the stack fumigated with 120 tablets. However, it was later determined that the live larva was from an untreated control. Less than 5 percent of the larvae held as controls were dead.

The number of pink bollworm larvae found in the bolls from each test location averaged 13.6. All larvae were dead. The same average held for larvae in the cotton bolls retained as controls and all were alive. No moths emerged from the cotton bolls exposed to the fumigant; 22 emerged from the bolls held as controls.

The phosphine gas (PH<sub>3</sub>) concentrations during fumigation are given in table 1. Exceptionally

TABLE 1.—*Phosphine concentrations for various dosage rates at 12 locations in 5 stacks of cottonseed when fumigated for different periods at Mesa, Ariz., 1968*

| Tablets per 1,000<br>cubic feet | Phosphine concentrations after— |               |               |               |               |
|---------------------------------|---------------------------------|---------------|---------------|---------------|---------------|
|                                 | 12 hours                        | 24 hours      | 48 hours      | 72 hours      | 96 hours      |
|                                 | <i>P.p.m.</i>                   | <i>P.p.m.</i> | <i>P.p.m.</i> | <i>P.p.m.</i> | <i>P.p.m.</i> |
| 60 -----                        | 124-168                         | 255-292       | 467-554       | 467-685       | 270-503       |
| 90 -----                        | 131-146                         | 255-299       | 467-481       | 561-612       | 343-547       |
| 120 -----                       | 175-401                         | 503-569       | 919-1,050     | 1,188-1,298   | 984-1,079     |
| 150 -----                       | 306-401                         | 605-663       | 1,021-1,239   | 1,400-1,516   | 1,225-1,400   |
| 180 -----                       | 328-350                         | 656-714       | 1,123-1,254   | 1,560-1,677   | 1,531-1,633   |

uniform gas distribution is shown. Peak concentrations were obtained at 72 hours in each stack. Because of low relative humidity under the tarps, some aluminum phosphide tablets were not completely decomposed when the tarps were removed from the stacks.

## Test 2

### Materials and Methods

Since one larva was thought to have survived the 120-tablet dosage in test 1, it was decided to fumigate the cottonseed in a second test with 180 tablets per 1,000 cubic feet and expose the seed to the fumigant for 120 hours. Also, considering the limited number of tests conducted, a safety factor of 60 additional tablets would be needed to assure positive control for quarantine use. The exposure time was increased from 96 to 120 hours because all the aluminum phosphide tablets had not completely decomposed at 96 hours in test 1.

The procedure used in the first test was repeated in the second test except 4-mil polyethylene rather than 6-mil nylon tarp was used to cover the stacks of cottonseed, and gas samples were drawn from only one stack.

At the start of the test the temperature of the cottonseed was 59° F. and the relative humidity in the warehouse was 62 percent. During exposure the warehouse temperature ranged from 45° to 66°. The temperature within the tarped stacks ranged from 52° to 58° and the relative humidity from 50 to 62 percent.

After 120 hours of exposure the stacks were uncovered and the building was aerated by natural draft for about 30 minutes. The test insects were recovered and mortality counts were made.

### Results

All insects exposed to the fumigant in each of the five stacks of cottonseed were dead. Thirty infested bolls from one stack and 28 from a second stack were examined, and 33 and 42 dead larvae, respectively, were found. Thirty bolls held as a control were examined and 41 live larvae were found. The remaining infested cotton bolls from the fumigated stacks of cottonseed and the controls were handled in the same manner as in the first test. No adults emerged from the fumigated bolls, but 44 pink bollworm moths emerged from the controls.

The range of phosphine concentrations at 12 locations in one cottonseed stack at the dosage of 180 tablets per 1,000 cubic feet for various exposures was as follows:

| <i>Exposure (hours)</i> | <i>Phosphine (p.p.m.)</i> |
|-------------------------|---------------------------|
| 24 -----                | 1,035-1,341               |
| 48 -----                | 1,837-2,304               |
| 72 -----                | 2,172-2,916               |
| 96 -----                | 2,100-2,872               |
| 120 -----               | 1,502-2,522               |

### Discussion

Phosphine was excellently distributed in test 2. As in test 1, peak concentrations were reached at 72 hours after fumigation. Higher concentrations were obtained in the second test than in the first at the 180 dosage rate because the relative humidity under the tarps was almost twice as high in the second test.

Samples of cottonseed were taken from each of the stacks both before fumigation and after the stacks had been fumigated twice and sent to a commercial seed-testing laboratory for germination tests. The results of these tests showed that

two fumigations with 180 aluminum phosphide tablets per 1,000 cubic feet had no effect on the germination of the cottonseed.

These tests showed that phosphine evolving from aluminum phosphide tablets is effective against pink bollworm larvae in covered stacks of cottonseed if the dosage rate is 180 tablets per 1,000 cubic feet for 120 hours and the relative humidity is at least 30 percent when the stacks are tarped.

As a result of these tests, the Plant Protection Division accepted a recommendation made by the Stored-Product Insects Research Branch, Mar-

ket Quality Research Division, for use of 180 aluminum phosphide tablets per 1,000 cubic feet in pink bollworm quarantine areas. The regulation stated, however, that the cottonseed should be in cotton fiber bags and covered with a 4- or 6-mil polyethylene or nylon tarp.

The following questions remained to be answered: Will phosphine penetrate multiwalled cottonseed shipping bags? Is it possible to use a lighter tarp? Is a dosage of 180 aluminum phosphide tablets per 1,000 cubic feet necessary to obtain 100-percent mortality?

## TESTS IN KANSAS

Immediately following the Arizona tests, exploratory tests were conducted to determine the permeability of phosphine gas ( $\text{PH}_3$ ) through various types of cottonseed shipping bags and to compare the  $\text{PH}_3$  retention value of 1.5-mil and 6-mil polyethylene tarps.

The tests were conducted in an Agricultural Stabilization and Conservation Service quonset at Waterville, Kans., where 10- by 10- by 10-foot frames of 2- by 4- inch lumber were used as fumigation chambers.

### Test 1

#### Materials and Methods

Six bags of differing construction as used by seed companies were filled with flame-delinted cottonseed and stitched shut; 10 bags were filled with crumpled newspapers and stitched shut; and 10 bags were filled with crumpled newspapers, stitched shut, and then all seams covered with masking tape. Two replacement tubeless tire valves were installed in a sidewall of each bag used in the test so that gas samples could be taken from inside the bags with a rotor pump without creating a vacuum or pressure in the bags. All the bags were then placed on pallets in the 1,000-cubic-foot chamber and covered with 6-mil polyethylene tarp. Polyethylene tubing was attached to the two valves on each bag, brought to the outside, and connected with a rubber nipple. A gas sampling line and a return line were placed above the shipping bags in the fumigation chamber and also in an empty chamber,

which was covered with 1.5-mil polyethylene tarp.

Twenty aluminum phosphide tablets were placed under the pallets, and sand snakes were placed around the chamber to hold the 6-mil polyethylene tarp down. Twenty aluminum phosphide tablets were also placed in the empty chamber, and the 1.5-mil polyethylene tarp was sand snaked down. Gas samples were drawn from each fumigation chamber at 24, 48, 72, 96, and 120 hours after the fumigant was applied. Concentrations of  $\text{PH}_3$  were determined by gas chromatography.

During the test the temperature varied from 56° to 88° F. and the relative humidity from 40 to 85 percent. The temperature was 77° and the relative humidity 48 percent when the fumigant was applied.

#### Results

Table 2 describes bag construction and phosphine gas ( $\text{PH}_3$ ) concentrations found inside the tested bags. Average concentrations of gas in the shipping bags are compared in table 3.

The test results show that  $\text{PH}_3$  will penetrate multiwalled shipping bags even though they have polyethylene liners. The day-to-day concentrations found in the shipping bags varied to some extent. When the average concentrations were calculated (table 3), they showed very little difference as to type of bag or whether the bag contained cottonseed or crumpled newspaper. The 4-ply paper bag with polyethylene liner and the 4-mil polyethylene bag both filled with cottonseed

TABLE 2.—*Phosphine concentrations in various types of cottonseed shipping bags with different contents and in space above tarp in both chambers when bags were fumigated under 6-mil polyethylene tarp with 20 aluminum phosphide tablets per 1,000 cubic feet for different periods, Waterville, Kans., 1968*

| Content and type<br>of bag or<br>tarp <sup>1</sup> | Phosphine concentrations after— |        |        |        |        |
|--|---------------------------------|--------|--------|--------|--------|
|  | 24                              | 48     | 72     | 96     | 120    |
|  | hours                           | hours  | hours  | hours  | hours  |
|  | P.p.m.                          | P.p.m. | P.p.m. | P.p.m. | P.p.m. |
| COTTONSEED   |                                 |        |        |        |        |
| 5-ply paper, inner ply plasticized ---             | 215                             | 205    | 294    | 231    | 185    |
| 5-ply paper -----                                  | 364                             | 404    | 340    | 343    | 323    |
| 4-ply paper plus polyethylene liner -              | 109                             | 213    | 248    | 185    | 198    |
| 4-ply paper, 3 plies perforated -----              | 273                             | 393    | 354    | 271    | 264    |
| 4-mil polyethylene bag -----                       | 38                              | 142    | 172    | 125    | 125    |
| Poly-Weve -----                                    | 354                             | 349    | 392    | 330    | 290    |
| CRUMPLED NEWSPAPER                                 |                                 |        |        |        |        |
| 5-ply paper, inner ply plasticized --              | 295                             | 404    | 385    | 304    | 297    |
| 5-ply paper -----                                  | 372                             | 448    | 392    | 337    | 290    |
| 5-ply paper plus polyethylene liner -              | 191                             | 404    | 420    | 343    | 304    |
| 4-ply paper plus polyethylene liner -              | 104                             | 393    | 357    | 317    | 317    |
| 4-ply paper, 3 plies perforated <sup>2</sup> ----  | 295                             | 426    | 431    | 330    | 297    |
| 4-mil polyethylene bag -----                       | 43                              | 197    | 280    | 277    | 284    |
| Poly-Weve -----                                    | 369                             | 448    | 403    | 310    | 297    |
| 3-ply paper plus polyethylene liner -              | 199                             | 404    | 413    | 290    | 310    |
| 4-ply paper, 3 plies perforated <sup>2</sup> ----  | 328                             | 470    | 383    | 337    | 304    |
| 4-ply paper, asphalt between 2 plies               | 261                             | 437    | 396    | 330    | 310    |
| CRUMPLED NEWSPAPER<br>ALL SEAMS TAPED              |                                 |        |        |        |        |
| 5-ply paper, inner ply plasticized ---             | 187                             | 339    | 396    | 337    | 290    |
| 5-ply paper -----                                  | 340                             | 459    | 396    | 330    | 310    |
| 5-ply paper plus polyethylene liner -              | 162                             | 404    | 396    | 337    | 330    |
| 4-ply paper plus polyethylene liner -              | 66                              | 251    | 330    | 231    | 251    |
| 4-ply paper, 3 plies perforated <sup>2</sup> ----  | 317                             | 470    | 389    | 297    | 297    |
| 4-mil polyethylene bag -----                       | 66                              | 207    | 284    | 310    | 304    |
| Poly-Weve -----                                    | 328                             | 420    | 370    | 343    | 310    |
| 3-ply paper plus polyethylene liner -              | 109                             | 294    | 337    | 343    | 330    |
| 4-ply paper, 3 plies perforated <sup>2</sup> ----  | 371                             | 420    | 350    | 304    | 310    |
| 4-ply paper, asphalt between 2 plies               | 262                             | 364    | 403    | 350    | 323    |
| 6-mil polyethylene tarp -----                      | 360                             | 462    | 383    | 330    | 317    |
| 1.5-mil polyethylene tarp -----                    | 306                             | 399    | 271    | 264    | 251    |

<sup>1</sup> All bags stitched shut.

<sup>2</sup> Similar bags but supplied by different manufacturers.

TABLE 3.—Average phosphine concentrations in various types of cottonseed shipping bags with different contents and in space above tarp in both chambers when bags were fumigated under 6-mil polyethylene tarp with 20 aluminum phosphide tablets per 1,000 cubic feet for different periods, Waterville, Kans., 1968

| Type of bag or tarp                                | Bag content <sup>1</sup> |                    |                                      |
|--|--------------------------|--------------------|--------------------------------------|
|  | Cottonseed               | Crumpled newspaper | Crumpled newspaper (all seams taped) |
|  | P.p.m.                   | P.p.m.             | P.p.m.                               |
| 5-ply paper, inner ply plasticized -----           | 244                      | 337                | 310                                  |
| 5-ply paper -----                                  | 355                      | 368                | 369                                  |
| 4-ply paper plus polyethylene liner -              | 192                      | 298                | 226                                  |
| 4-ply paper, 3 plies perforated <sup>2</sup> ----- | 311                      | 356                | 354                                  |
| 4-mil polyethylene bag -----                       | 120                      | 216                | 234                                  |
| Poly-Weve -----                                    | 343                      | 365                | 354                                  |
| 5-ply paper plus polyethylene liner -              | ---                      | 332                | 326                                  |
| 3-ply paper plus polyethylene liner -              | ---                      | 323                | 285                                  |
| 4-ply paper, 3 plies perforated <sup>2</sup> ----- | ---                      | 364                | 351                                  |
| 4-ply paper, asphalt between 2 plies ---           | ---                      | 347                | 340                                  |
| 6-mil polyethylene tarp -----                      | 370                      | ---                | ---                                  |
| 1.5-mil polyethylene tarp -----                    | 298                      | ---                | ---                                  |

<sup>1</sup> All bags stitched shut.

<sup>2</sup> Similar bags but supplied by different manufacturers.

showed some resistance to the penetration of  $\text{PH}_3$ . Placing masking tape over the stitched seams of the bags did not prevent the penetration of gas.

At the end of table 2 the concentrations of  $\text{PH}_3$  found in the chambers covered with 6-mil and 1.5-mil polyethylene tarps are compared. The 1.5-mil tarp compared favorably with the 6-mil tarp in retention value. However, the 1.5-mil tarp had to be handled with additional care to prevent tearing or punching holes in it.

## Test 2

### Materials and Methods

Another test was conducted at Waterville in the 10- by 10- by 10-foot fumigation chambers, which were covered with 6-mil polyethylene tarp. Twenty-five laboratory-reared pink bollworm larvae were placed in each of three empty chambers. The dosage rate was two aluminum phosphide tablets per 1,000 cubic feet and exposure time was 120 hours. The concentrations of  $\text{PH}_3$  ranged from 23 to 30 p.p.m. during exposure. The temperature in the fumigation chambers ranged from 59° to 83° F. during the test and the relative humidity from 60 to 80 percent.

### Results

All larvae were killed in the 120-hour exposure.

### Discussion

The results obtained in the Waterville study had to be tested in the field before a quarantine treatment could be recommended to the Plant Protection Division.

## TESTS IN OKLAHOMA

Cottonseed and warehouse facilities for fumigation tests were used at the Oklahoma Cotton Research Center at Chickasha.

Three types of shipping bags were selected for testing. They were (1) 4-ply paper bags with three plies perforated and the inside ply Capcote treated, that is, treated with a liquid polyethylene film that forms a moisture barrier; (2) 3-ply paper bags with a 1-mil polyethylene liner; and (3) 4-mil polyethylene liners for burlap bags.

These shipping bags were selected for the tests because they were thought to best represent those used by the cottonseed industry throughout the cotton-growing area of the United States.

To check the  $\text{PH}_3$  concentrations in the different bags during the tests, two replacement tubeless tire valves were installed, as described previously, in the shipping bags that would contain the pink bollworm-infested cotton bolls. These bags would be placed at the bottom, middle,



and top of the stack of bagged cottonseed to be fumigated.

### Test 1

#### Materials and Methods

The first test was conducted during November 1968. Laboratory-reared pink bollworm larvae from Brownsville, Tex., and pink bollworm larvae infesting cotton bolls in the field at Phoenix, Ariz., were used. The laboratory-reared larvae were placed in small plastic vials along with sterile cotton and some synthetic media. Perforated caps with a 40-mesh screen insert were used to confine the test insects in the vials and to afford ventilation. Thirty infested cotton bolls and one vial containing 10 laboratory-reared larvae were placed in a small cloth bag, which was closed with tie strings. The test insects were placed near the center of the cottonseed-filled shipping bags before they were closed with a portable stitcher.

During stacking, one each of the three types of shipping bags was placed at each of the following locations: Bottom, half way between bottom and middle, half way between middle and top, and top of the stack. Polyethylene tubing was attached to the valves in the bags placed at the bottom, middle, and top of the stack. Tubing was also placed at the bottom and near the top of the stack to take gas samples under the polyethylene tarp.

The stack was covered with 1.5-mil polyethylene tarp. Forty aluminum phosphide tablets were used to fumigate the stack of cottonseed-filled bags containing about 875 cubic feet. This was a dosage of about 45 tablets per 1,000 cubic feet. They were placed on brown paper at the base of the stack. Sand snakes were placed around the stack to seal the polyethylene tarp to the concrete floor.

When the fumigant was applied, the temperature was 66° F. and the relative humidity 52 percent. During the 120-hour exposure the temperature ranged from 44° to 66° and the relative humidity from 32 to 85 percent. The temperature of most of the fumigated cottonseed was 56°; however, several bags obtained from an outside bin would have averaged somewhat higher. The

moisture content of the cottonseed was unknown.

Gas samples were drawn at 24, 48, 72, 96, and 120 hours after the fumigant had been applied.

The 1.5-mil polyethylene tarp was removed from the stack after 120 hours' exposure. The aluminum phosphide tablet residue was removed from the warehouse and the warehouse aerated for 30 minutes before the stack was taken down and the test insects were recovered from the shipping bags.

The test insects and gas samples were sent to the Mid-West Grain Insects Investigations laboratory at Manhattan, Kans., where the gas samples were analyzed for  $\text{PH}_3$  by chromatography and the laboratory-reared pink bollworm larvae were examined for mortality. The infested cotton bolls were shipped to the Western Cotton Insects Investigations laboratory at Phoenix, Ariz., for incubation.

#### Results

In this test 100 percent of the laboratory-reared pink bollworm larvae were killed and 2 percent of the controls were cannibalized. The Phoenix laboratory was unsuccessful in breaking diapause of insects in the infested bolls and consequently each boll was dissected and each seed was cut and examined. The number of dead larvae averaged 7.6 in each group of cotton bolls that had been fumigated in each shipping bag. The larvae found alive averaged eight in each group of bolls held as controls. Only two dead larvae were found in the controls.

Phosphine concentrations during the test are shown in table 4.

Table 4 shows that gas concentrations peaked at 72 hours. The  $\text{PH}_3$  concentrations in the two types of multiwalled paper bags were about the same in the three locations at each sampling time. The 4-mil polyethylene bags resisted penetration to some extent; only about 50 percent of the concentration under the polyethylene tarp was found in the bags.

Although 100-percent mortality of the pink bollworm larvae was obtained with 45 aluminum phosphide tablets per 1,000 cubic feet, this dosage was the minimum and left little tolerance for human error.

TABLE 4.—*Phosphine concentrations in and around cottonseed shipping bags fumigated under 1.5-mil polyethylene tarp with 45 and 60 aluminum phosphide tablets per 1,000 cubic feet for different periods, Chickasha, Okla., 1968 and 1969*

| Dosage, location<br>of bag or<br>airspace, and<br>bag identity | Phosphine concentrations after— |               |               |               |               |
|--|---------------------------------|---------------|---------------|---------------|---------------|
|  | 24<br>hours                     | 48<br>hours   | 72<br>hours   | 96<br>hours   | 120<br>hours  |
| 45 TABLETS   | <i>P.p.m.</i>                   | <i>P.p.m.</i> | <i>P.p.m.</i> | <i>P.p.m.</i> | <i>P.p.m.</i> |
| Bottom:  |                                 |               |               |               |               |
| Acala—Capcote treated .....                                    | 218                             | 437           | 468           | 390           | 265           |
| Growers—3-ply polyethylene<br>liner .....                      | 203                             | 437           | 468           | 390           | 265           |
| 4-mil polyethylene .....                                       | 55                              | 203           | 250           | 234           | 187           |
| Airspace .....   | 312                             | 484           | 499           | 421           | 281           |
| Middle:  |                                 |               |               |               |               |
| Acala—Capcote treated .....                                    | 218                             | 437           | 468           | 390           | 281           |
| Growers—3-ply polyethylene<br>liner .....                      | 203                             | 406           | 437           | 374           | 265           |
| 4-mil polyethylene .....                                       | 78                              | 156           | 203           | 234           | 203           |
| Top:   |                                 |               |               |               |               |
| Acala—Capcote treated .....                                    | 218                             | 452           | 484           | 390           | 296           |
| Growers—3-ply polyethylene<br>liner .....                      | 203                             | 437           | 468           | 374           | 265           |
| 4-mil polyethylene .....                                       | 39                              | 468           | 203           | 203           | 172           |
| Airspace .....   | 296                             | 499           | 515           | 406           | 234           |
| 60 TABLETS   |                                 |               |               |               |               |
| Bottom:  |                                 |               |               |               |               |
| Acala—Capcote treated .....                                    | 335                             | 670           | 830           | 830           | 660           |
| Growers—3-ply polyethylene<br>liner .....                      | 290                             | 620           | 800           | 770           | 650           |
| 4-mil polyethylene .....                                       | 35                              | 140           | 230           | 290           | 290           |
| Airspace .....   | 450                             | 820           | 910           | 820           | 690           |
| Middle:  |                                 |               |               |               |               |
| Acala—Capcote treated .....                                    | 330                             | 690           | 820           | 800           | 650           |
| Growers—3-ply polyethylene<br>liner .....                      | 330                             | 690           | 830           | 790           | 660           |
| 4-mil polyethylene .....                                       | 40                              | 150           | 220           | 280           | 290           |
| Top:   |                                 |               |               |               |               |
| Acala—Capcote treated .....                                    | 370                             | 730           | 860           | 810           | 670           |
| Growers—3-ply polyethylene<br>liner .....                      | 330                             | 720           | 840           | 800           | 670           |
| 4-mil polyethylene .....                                       | 30                              | 110           | 220           | 220           | 230           |
| Airspace .....   | 450                             | 830           | 920           | 830           | 680           |

## Test 2

### Materials and Methods

A second test was conducted in February 1969. The same types of shipping bags and the same procedure were used in the second test except the dosage rate was raised to 60 tablets per 1,000 cubic feet and the only test insects used were those infesting cotton bolls in the field. Also, the multiwalled shipping bags were closed with masking tape instead of the portable sack stitcher, and the 4-mil polyethylene bag liners were closed by gathering the top of each bag together and tying it with cotton cord.

The temperature of the cottonseed averaged 49° F. during fumigation. It was 42° and the relative humidity 60 percent when the fumigant was applied. The temperature ranged from 36° to 53° and the relative humidity from 30 to 85 percent during the test. The moisture content of the cottonseed was unknown.

After the cottonseed had been exposed 120 hours to the fumigant, the stack was dismantled after proper aeration, and the infested cotton bolls were recovered. The gas samples and test insects were sent to Manhattan, Kans. The gas samples were analyzed by chromatography. The infested cotton bolls were dissected and each seed was cut and examined for pink bollworm larvae.

### Results

In this test 100-percent mortality of pink bollworm larvae was obtained. The number of dead larvae averaged 10 in each group of 30 cotton bolls exposed to the fumigant. The larvae found alive averaged 11.8 in each group of infested bolls held as controls. Only three dead larvae were found in the controls.

Phosphine concentrations during the test are shown in table 4.

### Discussion

Little difference in  $\text{PH}_3$  concentrations was found in the two types of multiwalled paper shipping bags. Higher concentrations during the second test were due to the higher dosage as was expected. The  $\text{PH}_3$  concentrations in the 4-mil polyethylene bags in the second test were about the same as in the first test at a lower dosage. Concentration of gas continued to build up in the 4-mil polyethylene bags until the test was terminated.

As a result of these tests, the Plant Protection Division revised a regulation regarding the fumigation of cottonseed in pink bollworm quarantine areas. Cottonseed in multiwalled paper shipping bags may be fumigated with 60 aluminum phosphide tablets per 1,000 cubic feet under 1.5-mil polyethylene tarp. Thicker polyethylene may be used. Burlap bags with 4-mil polyethylene liners may be fumigated in the same manner if the liners are perforated.